Analytical Methods

Grain Size

SOP PTL-10-200

1.0 PURPOSE

1.1. The purpose of this document is to detail the standard operating procedure for the Malvern Mastersizer 2000 laser diffractor on a wet basis.

2.0 SCOPE

2.1. Applies to all laboratory staff who analyzes samples on the Mastersizer 2000.

3.0 RESPONSIBLE PARTIES

3.1. Lab Management
3.2. Laboratory Analysts

4.0 ASSOCIATED MATERIALS

4.1. PV Mastersizer 2000 Form (PTL-10-200A)
4.2. TM Mastersizer 2000 Form (PTL-10-200C)
4.3. PR Mastersizer 2000 Form (PTL-10-200D)
4.4. Instrument Use Log (PTL-12-010A)
4.5. Routine Maintenance Form (PTL-12-011A)
4.6. Non-Routine Maintenance Form (PTL-12-012A)

5.0 INSTRUMENTATION

5.1 The Mastersizer 2000 is a laser diffractor that measures the particle size of liquids and powder in a wet suspension on a volume basis. The system is comprised of the following:

a. Mastersizer 2000
b. Hydro 2000S Sample Dispersion Unit
c. Flow cell
d. Computer
e. Printer
f. Solvent waste container

5.2. The Mastersizer 2000, in conjunction with the computer, measures the particle size of the sample that passes in front of the laser and

in turn produces a volume-weighted histogram and numerical statistics associated with the sample type.

5.3. The flow cell is fitted into the slot of the Mastersizer 2000.

5.4. The flow cell is connected with tubing to the Hydro 2000S sample dispersion unit. The Hydro 2000S recirculates the dispersed sample suspension through the flow cell at a user set speed. The sample suspension circulates through the flow cell as the laser beam passes through the flow cell and diffracts and scatters light.

6.0 PERFORMANCE VERIFICATIONS

6.1. Standards of a known size are analyzed quarterly by a trained operator to ensure that the Mastersizer 2000 is within normal operating parameters.

6.2. The standard is prepared and analyzed according to individual test methods created for each standard. These methods reflect the standard manufacturer's recommendations for sample prep and/or analysis and are stored in the active lab method binder.

6.3. Performance Verification (PV) forms, to be completed by the analyst, describe the standard used, results, pass or fail and analyst and reviewer signature.

6.4. The performance verification passes if the mean/median size is +/- 3% of the known value for all reference materials.

6.5. The data and PV form are in the laboratory maintenance binders.

7.0 USE LOG

7.1. The Use Log form is maintained in the maintenance binder. The log documents all project numbers, the user, date, and company name.

8.0 PROJECT RECORD

8.1 Record all work on PR form

9.0 INSTRUMENT SET-UP

9.1. Turn computer and optical unit on. To turn on the optical unit, press the button on the side of the instrument.

9.2. Fit tubing to recirculator (Hydro2000S) and flow cell.

9.3. Fit the flow cell into the slot and lock it in place by turning the handle.

9.4 To turn on the Hydro 2000S press the button on the back of the recirculator, located right above the power cord.

9.5 Make sure the waste tubing in the back of the instrument is in the solvent waste container.

10.0 SOFTWARE SET-UP

10.1. Open Mastersizer 2000 program. On main windows screen, select
Start
All Programs
Malvern Instruments
Mastersizer 2000 Version 5.1

Instead of this you can also click on the Mastersizer 2000 Icon on the desktop.

10.2 A window comes up asking you to type your user name to log on. The default user name is: Administrator. Press **OK** to go on.

10.3 Another window comes up with four choices
a) Run an existing SOP
b) Edit an existing SOP
c) Use the SOP Creation Wizard
d) Make manual Measurement

Depending on what you want to do select one and press **OK**.

11.0 NEW SOP CREATION WIZARD

11.1 Window opens up – Hit **Next**

11.2 Chose the sample handling unit – Hydro 2000S (A)

11.3 Hit **Next**

11.4 Enter information for Material Name by either choosing:
a) from dropdown menu
or if not already present
b) hitting the material button
1) Now pick from list of assorted materials with known refractive indexes and hit move (that brings it into the dropdown menu on the previous window)
2) Otherwise hit add and fill in the
a) Name of sample
b) Refractive Index of Sample
c) Adsorption
d) Density (Default is 1)

There is a box that gives you a choice of different values for blue light. Our default is to use the same for red and blue light.

11.5. Hit **OK**

11.6 Now info is in the sample materials box which is really the dropdown menu on the other page.

11.7 Hit **OK**

11.8. Enter the dispersant (aka carrier) by choosing from the dropdown menu or if it is not there by hitting the **Dispersant** button and filling in:
a) Name of dispersant
b) Refractive Index of dispersant
c) Adsorption
d) Density (Default is 1)

11.9. Hit **OK**

11.10. Under Result Calculation hit the **Models** button and pick appropriate model:
a) General Purpose (our default)
b) Multiple narrow modes

c) Single mode

11.11. Under Calculation sensitivity choose enhanced normal.

11.12. For particle shape choose:
a) irregular (default)
b) spherical

11.13 Hit **OK**

11.14 Hit **Next**

11.15 Labels window opens fill in appropriate areas
1) Sample name
2) Source – choose supplier from dropdown menu
3) Source name – enter client name
4) Bulk Lot type – choose Lot # from dropdown menu
5) Bulk lot ref. – leave blank (Lot # to be entered at time of analysis for each sample)

11.16 Operator Instructions and comments:
1) check box Display instructions before measurement
2) check box Allow operator to enter comments before measurement
3) uncheck box display instructions after measurement

11.17 Hit **Next**

11.18 Report/Savings Window opens up
a) do not check box always print results (we print them later as needed)
b) under reports page pick Result Analysis PTL –Wet(M).pag (our default) from dropdown menu

11.19 Hit next

11.20 Measurement Window opens up
a) Fill in measurement time (12 Seconds is the default)
b) Fill in background time (12 Seconds is the default) Background time needs to be the same or longer than the measurement time

11.21 Hit **Advanced Options** button and fill in:
- a) Obscuration limits (10-20 is the default) If that needs to be changed uncheck the box and fill in the appropriate limits.
- b) Obscuration filtering – check box enable filtering (this makes sure only measurements in the obscuration limits are made)
- c) For large particles go to Red Only Measurement and check the box enable red only measurement

11.22 Hit **OK**

11.23 **Hit Next**

11.24 Under Sampler Settings set the pump/stir speed to the appropriate RPMs (2000 RPM is our default)

11.25 Under ultrasonics chose from different settings (default is none)

11.26 Under Tank Fill choose manual

11.27 Hit **Next**

11.28 Under check box "create average results" Measurement Cycles pick:
- a) Aliquots per SOP (1 is our default)
- b) Measurements per aliquot (4 is our default)
- c) Delay (60 Seconds is our default)

11.29 Cleaning – choose manual
Make sure to uncheck box "Unable after each aliquot"

11.30 Hit **Next**

11.31 Quantities (default on all choices is either don't specify or none)

11.32 Hit **Next**

11.33 Hit **Finish**

11.34 Save SOP as company name – sample name

Info: If you're ever in doubt about what to choose on any of these hit one of the advice buttons located on every page of the SOP wizard or ask another analyst.

12.0 MANUAL MEASUREMENT

12.1 Open **File**

12.2 Select **New**

12.3. Enter File name (PTL project number; i.e.7731)

12.4. Hit save

12.5 Go to Measure – Manual

12.6 Under Measurement Options Window choose:
a) Materials and fill in as shown in 7.4
b) Measurement fill in everything like 7.19
c) Measurement Cycles fill in number of cycles (measurements) as well as the delay (60 Seconds)

12.7 Hit **OK**

12.8 A screen opens up – when system is clean and ready for measurement

12.9 Hit **Start**

12.10 Once done it goes through the alignment and background measurement screens

12.11 When you get to the documentation screen, enter lot number as well as pre-measurement instructions:
a) Customer name: Sample name:
b) Per Test Method:
c) Analyst:
d) Particle Technology Labs, Ltd. PTL ID: five digit number

12.12 Hit **OK**

12.13 Add sample until appropriate obscuration is reached (in the green range)

12.14 Hit **Start**

13.0 MEASURING USING AN SOP

13.1. Go to File – New

13.2. Enter File name (PTL project number; i.e.7731)

13.3. Hit save

13.4. Go to Measure – start SOP

13.5 Fill the appropriate SOP from list and hit open

13.6 Full recirculator with appropriate solvent

13.7 It goes through the aligning as well as the background measuring screen

13.8 Documentation Screen opens up

13.9 Enter the lot number as well as pre-measurement instructions:

a) Customer name: Sample name:
b) Per Test Method:
c) Analyst:
d) Particle Technology Labs, Ltd. PTL ID: Five digit number

13.10 Hit **OK**

13.11 Add sample until the appropriate obscuration is reached.

13.12 Hit **Start** (that starts the analysis)

13.13 Once done window will open up asking you if you want to run the analysis again

13.14 Hit No after cleaning system follow steps 13.5-13.13.

14.0 CLEANING WINDOWS

14.1. Remove the flow cell from the housing.

14.2. Place flow cell on a paper towel.

14.3. Using the Malvern screwdriver, remove the windows from the flow cell and wipe the surface with cleaning fluid (ex: Acetone, Isopropyl Alcohol, glass cleaner) and a Kimwipe. NEVER wipe the windows when they are dry.

14.4. Check that there are not any fingerprints or smudges on the windows.

14.5. If the windows appear clean, reassemble the flow cell.

14.6. Place flow cell back into the optical unit.

14.7. Perform a background to ensure that the cell is clean and within operating requirements.

14.8 After changing carrier fluids or prior to each new project, a background is performed and printed. The printed background will be placed in the project file.

15.0 PRINTING DATA

15.1. Go to file – open and open the desired project file.

15.2. That opens the record window of the file.

15.3. The average of all runs will be printed and given to the client unless otherwise justified. In addition individual runs can be released at client 's request.

15.4 Select Result Analysis PTL – Wet(M) from toolbar unless otherwise specified.

15.5 That brings up the desired run in the default printout view.

15.6. Hit the printer icon in the toolbar to print out results.

15.7. Select OK.

16.0 PRINTING BACKGROUND

16.1. A background will be printed for the following circumstances:

16.1.1. One background will be printed for each sample or set of samples in a project, which are analyzed without interruption. When a stop in the analysis, break, or end of the day occurs, a new background will be printed to verify that those subsequent samples were analyzed under the same background integrity.

16.1.2. When the carrier fluid is switched. If a project contains samples that need to be analyzed in more than one type of carrier fluid, a background will be printed for each carrier fluid type.

16.1.3. For a performance verification.

16.2 The background is saved with each measurement and can be called back after the analysis.

16.3 Under File, select Open.

16.4 Choose the file that is needed.

16.5 Select the record number or the analysis that you would like to retrieve the background from.

16.6 Click on Data (M) in bar located under the tool menus or go to Configure, View/Report Selection and mark the box next to Data (M). Hit OK.

16.7 Select the print button from the tool bar to print the background.

16.7 Place the background in the project file or with the performance verification.

17.0 CLEAN UP

17.1. Rinse clean carrier fluid through the system until the background is clean.

17.2. If necessary clean the windows and rinse again. (See section 14 Cleaning Windows)

18.0 INSTRUMENT SHUT DOWN

18.1. Exit the Malvern Mastersizer 2000 program

18.2 Turn off the instrument on the side by pressing the button to the off position and pressing the button on the back of the Hydro 2000S to the off position.

18.3 Power the computer down.

19.0 ROUTINE MAINTENANCE

19.1. The connecting tubing and O-rings holding in the windows are checked and/ or replaced quarterly. The documentation concerning the replacement is found in the instrument maintenance binder.

19.2 To change the tubing, first drain the system of all contents. Then remove the tubing from the flow cell by gently pulling the tubing off. The tubing is then removed from the Hydro 2000S by gently pulling. Next, cut two new pieces of tubing the same lengths as the tubing, which was removed. Attach the new tubing to the recirculator and then to the flow cell. Verify that the tubing was placed in the proper locations for circulation.

The tubing will be replaced every quarter.

19.3 To change the O-rings, first drain the system of all contents. Next, remove the cell from the instrument and place on a clean paper towel. Using the Malvern tool, remove the windows from the cell. Very gently, push the windows out of the holder over the paper towel. It is important to keep the windows close to the table top so that they do not fall and break. Once the windows have been removed, use your finger to remove the O-rings. These can be thrown away. Place the new O-rings around the windows and very carefully and gently place them into the window holder. Using as little force as possible, press the O-rings and windows into the window holder.

When O-rings and windows are secure in window holder, replace into cell with the aide of the Malvern tool.

20.0 NON-ROUTINE MAINTENANCE

If the instrument is having any problems, the following steps are taken.

20.1 Ensure that the Performance Verification is current.

20.2 Check the settings on the instrument. Are they the normal set-up?

20.3 If a problem is encountered, notify laboratory management.

20.4 If it is determined that the instrument should be designated as is Out of Service, place a sign on the instrument which clearly states "Out of Service" and the date this occurred.

20.5 Contact the instrument manufacturer service technician.

20.6 If the instrument is repaired in-house, then a Performance Verification will be run to get it back IN SERVICE.

20.7 If the instrument is shipped to the manufacturer for repair, then upon the return of the instrument it will be re-qualified.

21.0 ADDITIONAL INFORMATION

21.1. Consulting the manufacturer's manual is very important for optical theory and operation instructions. This is a procedure guide written to be followed as an adjunct to the PTL SOP.

21.2. Malvern support. Malvern has a technical service department, which is very helpful.

22.0 APPROVALS

Originator _____ Date _____

Director or Laboratory Management _____ Date _____

Quality Assurance _____ Date _____

23.0 CHANGE OF HISTORY

Revision #	Date	Reason for Change	Require Training?
000	02-21-03	Original	
001	05-14-03	Revised section 18.1 and added the last statement into section18.2.	
002	09-24-04	Modified 6.4, 11.11,12.9 and 20.2. Added "Require Training?" into section 22. Added sections 23 and 24, "Documentation Review" and "Impact Assessment" respectively. Added "Effective Date" into heading.	Yes
003	12-13-04	Modified section 14.3.	Yes
004	05-11-05	Added sections 4.2, 4.3, 4.4, and 8.1. Modified- 11.18a from "check box always print results" to "do not check box always print results (we print them later as needed)." Section 11.28- added check box "create average results" Section 11.29- added "Make sure to uncheck box "Unable after each aliquot". Changed 13.14 to "Hit No after cleaning system follow steps 13.5-13.13.	Yes

24.0 DOCUMENTATION REVIEW

Review Date	Comments
05-11-05	See section 23.

25.0 IMPACT ASSESSMENT

SOPs #	Comments
None	
None- 12-13-04	
None- 05-11-05	